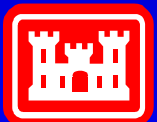


Pre-Contract Laboratory Evaluation

Dr. Douglas Taggart, Dr. Denise MacMillan,
and Dr. Trudy Olin-Estes

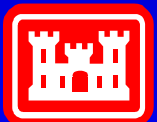


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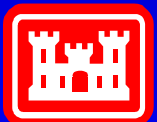
Presentation Organization

- Environmental Chemistry Branch: mission, resources, quality assurance activities
- Quality assurance activity for the Greens Bayou Sediment Delineation Plan: Pre-contract laboratory evaluation
- Other quality assurance evaluation tools: tape audits, selected data validation, project and method specific laboratory audits, project specific PE samples



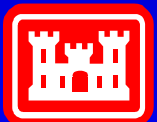
Mission

- Quality assurance: split sample quality assurance analysis, technical assistance, and problem solving to support USACE environmental HTRW programs
- Analytical chemistry support: water quality analysis and in-house HTRW project analysis
- Research: DOD relevant topics
- Research support: chemical analysis and chemistry technical support for ERDC research



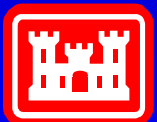
Environmental Chemistry Branch Resources

- Personnel: 29 Federal Government employees, 5 contract employees, 16 contract students
- Four team leaders: Dave Splichal, Prem Arora, Laura Percifield, and Bobby Jones
- Equipment: all major routine analytical equipment plus specialized equipment such as ICP/MS, LC/MS
- Excellent analytical chemistry facilities in two locations



ECB Quality Assurance Support Activities

- Technical support in analytical chemistry for USACE district personnel, HTRW CX chemists, ERDC research team members, and others
- Quality assurance split sample analysis and chemical quality assurance report preparation
- Laboratory audit support for the HTRW CX and USACE Districts
- Data evaluation, data validation, data assessment
- Analytical method modification and method development



Origin of Laboratory Evaluation Plan

- Dr. Trudy Olin-Estes (ERDC PI) contacted Dr. Denise MacMillan regarding laboratory selection for the Greens Bayou Sediment Delineation Project
- Technical concerns had been expressed regarding matrix interference problems based on previous analysis results
- Practical quantitation limits for the project were low for some analytes, additional analytes were requested for some parameters
- Agreement was made to complete the pre-contract award evaluation



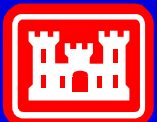
Laboratory Evaluation Plan

- Identify candidate laboratories based on input from project technical group
- Select analytical methods (VOA, BNA, Pest, PCBs, and metals) and establish reporting limits
- Contact laboratories with the proposed contracting procedure to include analysis of three project samples at no cost
- Collect representative composite samples
- Laboratories analyze samples and provide appropriate reports to the project team



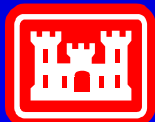
Laboratory Requirements

- Laboratories must analyze three representative project samples for VOA, BNA, organochlorine pesticides, PCBs, and metals at no cost
- Laboratories must provide a level 4 (CLP) quality control report both electronic and hard copy
- Laboratories may propose alternate analytical methods to meet data quality objectives



Evaluation Overview

- Raw data and reports for sample and batch quality control samples were evaluated for compliance, completeness, and correctness
- Evaluation of data package for each parameter completed by method expert at the ECB
- Evaluation criteria based on Greens Bayou SDP proposed laboratory evaluation program: SW-846 quality parameters; and the Corps “Shell for Analytical Chemistry Requirements”
- Data validation following functional guidelines to the extent possible



Example Validation Checklist

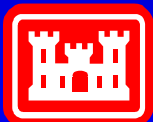
Data Verification and Validation Checklist - General
Environmental Chemistry Branch, Engineer Research and Development Center

Reviewer: _____ Method: _____ Project: _____

Today's Date: _____ Acceptance Criteria Reference: _____

(Check Correct Response. All "No" answers must be explained in the Comments Section Below.)

Review Item	Yes	No	NA
1. Do the chain-of-custody forms and the laboratory sample listing agree?			
2. Are the methods requested on the chains-of-custody the ones performed by the laboratory?			
3. Were the project-specific methods performed?			
4. Were samples properly preserved upon receipt at the laboratory?			
5. Were appropriate corrective actions performed, if warranted?			
6. Are trip blanks free of contamination?			
7. Are rinsate samples free of contamination?			
Identify samples affected by trip blank or rinsate contamination:			
8. Does the report narrative indicate any problems with the project?			
Comments/Recommendations			



Example Validation Checklist

Data Verification and Validation Checklist - Method 8270C
Environmental Chemistry Branch, Engineer Research and Development Center

Reviewer: _____ Project: _____

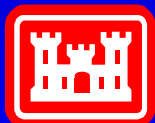
Today's Date: _____ Acceptance Criteria Reference: _____

Extraction Cycle: _____ Analytical Batch/Date: _____

Samples Reviewed: _____

(Check Correct Responses. All "No" answers must be explained in the Comments Section Below.)

Raw Data Review	Yes	No	NA
A. Initial Calibration			
1. Does the curve consist of at least five Calibration Standards?			
2. Is the low standard set at the LRL for each analyte?			
3. For all analytes, are the RSDs $\leq 15\%$, or $r \geq 0.995$ ($r^2 \geq 0.990$)?			
4. Does the ICV meet RSD $\leq 15\%$?			
Comments/Recommendations			
B. Tune Criteria / Continuing Calibration			
1. Are the OETPP mass criteria verified and acceptable every 12 hours?			
2. Is the OOT breakdown $\leq 20\%$?			
3. Are the benzidine and pentachlorophenol tailing factors acceptable?			
4. Is the Continuing Calibration Verification (CCV) standard run at the start of every 12 hours, and at the end of the analytical sequence?			
5. Are % Differences $\leq 20\%$ for the CCCs and project-specific target analytes?			
6. Are the RPs for the SPOCs ≥ 0.050 ?			
Comments/Recommendations			



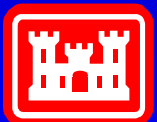
Data Validation Summary

- Three laboratories completed project samples and data packages for the selection process
- Laboratory A exhibited fewer and less severe deviations from method and project requirements (only Corps validated laboratory); deviations probably correctable
- Laboratory B had serious data deficiencies for some parameters
- Laboratory C had serious data deficiencies and submitted raw data with inappropriate manual integrations



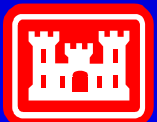
Laboratory A Data Validation Summary

- VOA: incomplete (four missing) target analyte list reported
- PCB: sample cleanup was not completed ; extraction log was not included; calibration was incomplete
- PEST: ICVs and CCVs incompletely reported; analyte list incomplete
- BNA: ICVs incomplete; breakdown and tailing not verification not reported
- Metals: ICV and CCV failures; prep blank contamination; spike recovery problems



Laboratory B Data Validation Summary

- VOA: no MDLs ; low standard four times PQL
- PCB: some peak enhancement; data package incomplete; no LD, MSD, or MS; other failures
- PEST: data package incomplete; initial calibration omitted; breakdown problems; other failures
- BNA: data package incomplete; calibration problems; internal standard failures; many other failures or omissions
- Metals: incorrect method

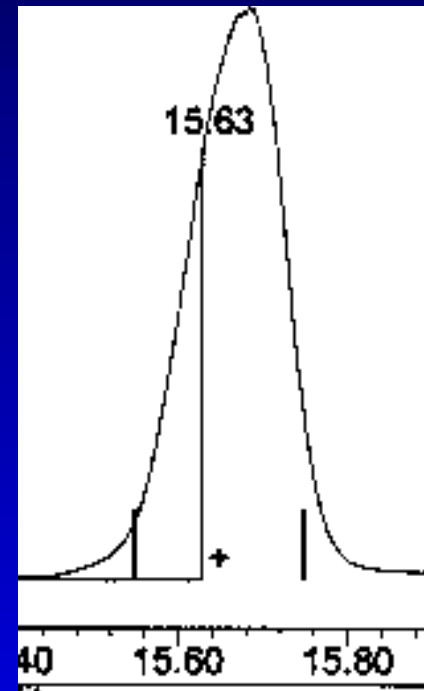
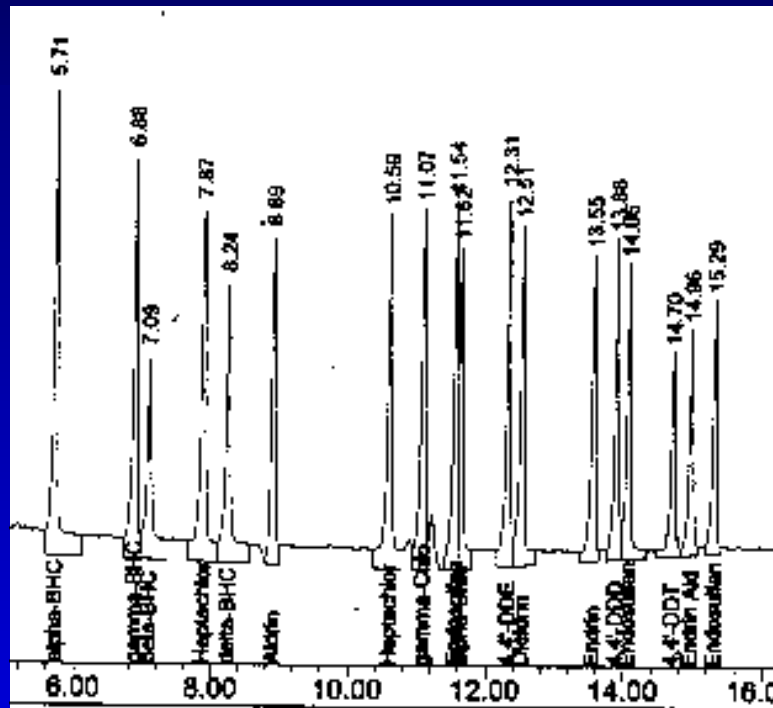


Laboratory C Data Validation Summary

- VOA: missed HT; analyte list incomplete
- PCB: improper peak integration; numerous other failures
- PEST: improper peak integration; numerous other failures
- BNA: ICV failures; MDLs not reported; target analyte list incomplete; proper corrective actions not followed ; other failures
- Metals: incorrect method; other QC failures



Inappropriate Manual Integration

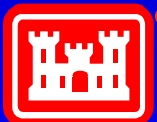


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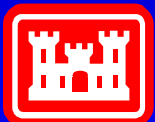
Conclusions of Pre-Contract Laboratory Evaluation

- Data validation was incredibly revealing of the potential ultimate product
- Process correctly and clearly indicated the current laboratory capability
- Process provided critical information as a starting point for negotiations with the contract laboratory
- Some laboratories apparently chose not to participate without payment for evaluation samples
- Process applies only to larger projects
- Past performance does not guarantee future performance



Other Quality Assurance Evaluation Tools

- Tape audits of project laboratories raw data - completed in conjunction with other data review efforts
- Selected data validation – targeted by project personnel for most critical project data
- Project and method specific laboratory audits – targeted audits by method expert
- Project specific PE samples – non-routine PE samples to be included with the project samples



Contacts

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- Ms. Laura J. Percifield, team leader, (402) 444-4314 (voice); (402) 341-5448 (fax); e-mail: Laura.J.Percifield@usace.army.mil
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